Quantification of ethyl acetate using FAIMS

Quantification of Ethyl Acetate using FAIMS

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1. Abstract

This work details an investigation into the quantification of ethyl acetate within wine using a Gas Chromatograph (GC) coupled with a Field Asymmetric Ion Mobility Spectrometer (FAIMS). The justification for an analytical approach to grading wine is presented along with how the GC-FAIMS technology may benefit the wine industry.

The quantification of ethyl acetate within a complex background such as wine also presented an excellent opportunity to investigate the capabilities of a FAIMS device as a GC detector.

It was found that the GC-FAIMS device was able to detect quantities of ethyl acetate well below the human threshold within a simple solvent. Detection of ethyl acetate within wine was found to be hindered due to the presence of ethanol. The lost sensitivity was recovered through increasing the pressure that the FAIMS sensor operated at.

2. Ethyl Acetate within Wine

Wine is enjoyed across the world where wine guides are often consulted before a purchase. The guides have become so important that the eventual price of a wine is affected by the classification provided [1]. It is therefore vital that both the industry and consumers have confidence with the grade given.

There have been numerous studies showing the deficiencies in judging wine via a panel. For example they can be susceptible to visual cues. Experts grading identical wines stated they had varying sweetness though the only difference between the wines was an odourless food dye [2,3]. They can also even fail to identify a unique wine from two other identical ones nearly one in three times [4,5]. This is not greatly surprising when the complexity of wine [6] and the difficulty that humans can experience isolating different scents [7] is considered. There is a justification to an analytical approach to inform the grading of wine.

While a full analytical approach to grading wine is beyond the ambition of this work the detection and quantification of ethyl acetate is pursued. The presence of ethyl acetate can add a desirable depth of body, richness and sweetness to a wine [8]. However, if abundance exceeds the human perception threshold limit of 100 - 200 mg/l [9,10] an aroma similar to acetone becomes prevalent and the wine is regarded as spoilt [9,10,11]. Some reviewers feel justified in disregarding a wine completely if any perception of ethyl acetate is detected.

The ability to quantify ethyl acetate within wine would enable the opportunity to better manage this multifarious compound in manufacture and throughout the product life cycle.

3. Apparatus and Experiment

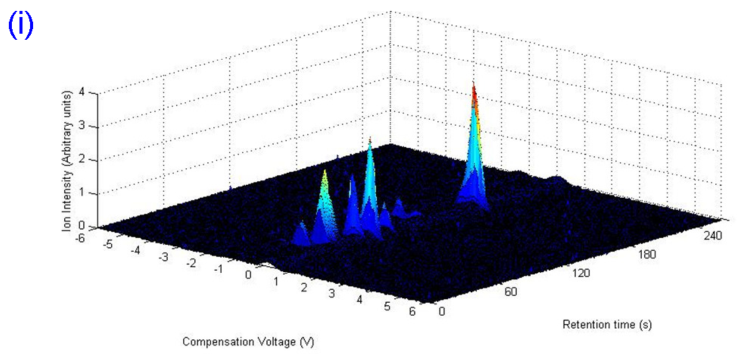
The GC-FAIMS system investigated was created through coupling a SRI 8610C with an Owlstone Tourist [12] incorporating a FAIMS sensor equivalent to that available within the Owlstone Lonestar [12]. The two separate units complement one another providing orthogonal data to one another. This provides greater separation of compounds which is ideal for investigating a particular compound within a complex background. The response of the system from wine is shown within **(i)**.

For those unfamiliar with FAIMS it is a technique capable of detecting trace quantities of analyte through the ionisation of a gaseous sample stream and filtering of resultant molecular-ions through the use of an electric field. The filtering can be adapted through modification of the imposed electric field so that it is possible to sweep across a range and detection of all the molecular-ions present is possible. FAIMS separates compounds due to their unique mobility within the environment of the sensor.

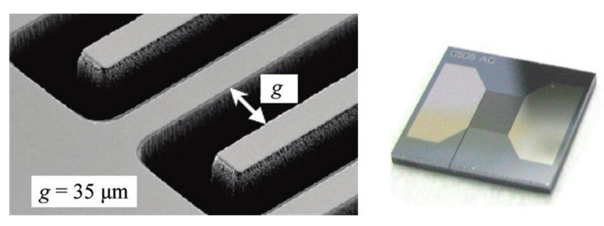
The Owlstone sensor

Integral to the complete system is the Owlstone FAIMS sensor. It is worthwhile discussing the sensor as it is unique as a FAIMS device due to the nanofabrication techniques employed in its manufacture. The result is a separation region consisting of 47 identical channels of 35 µm width **(ii)**. This geometry requires that the asymmetric waveform employed is run at a high frequency. The importance of this will be revealed within Section 5.

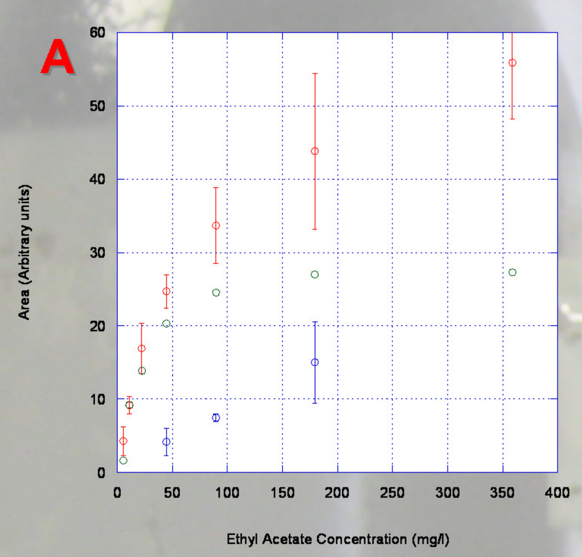
(i)



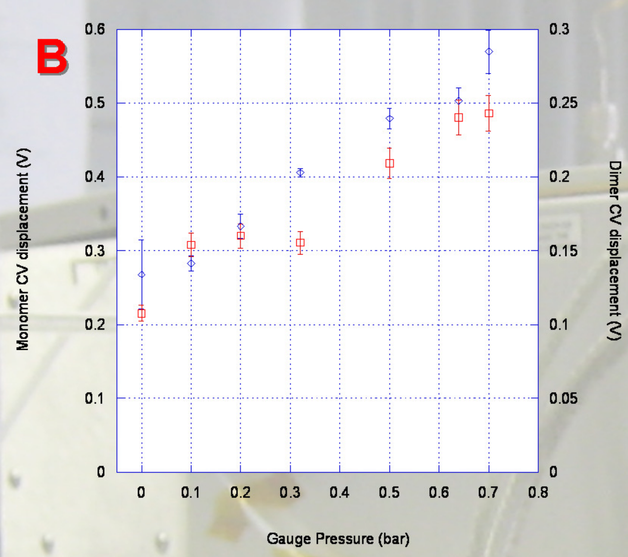
(ii)



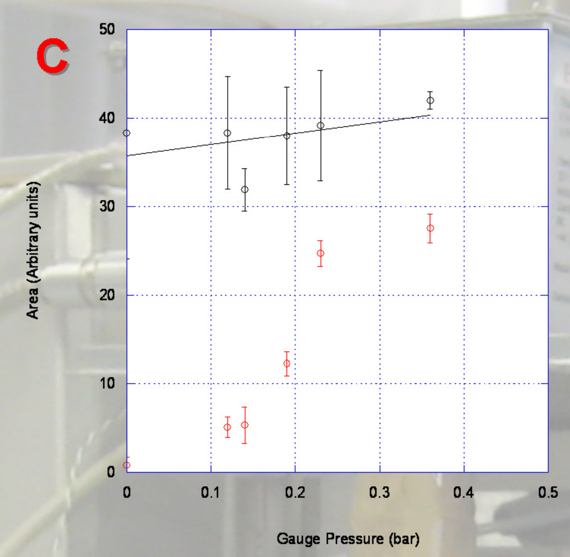
A



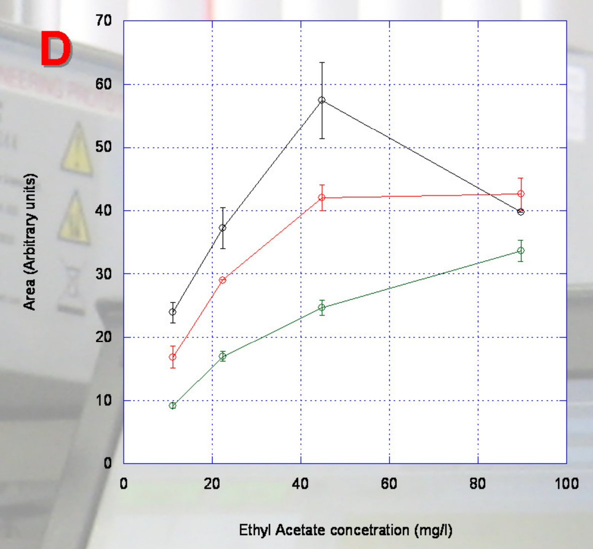
B



C



D



A: Distilled water spiked with ethyl acetate (**red**), wine spiked with ethyl acetate (**blue**) and distilled water and 12 % ethanol spiked with ethyl acetate using polar GC column (**green**).

B: Monomer (**blue**) and dimer (**red**) of ethyl acetate within solvent of distilled water at various pressures of carrier gas. Constant energy to ions.

C: Ethyl acetate within a solvent of distilled water (**black**) and solvent of distilled water and 12 % ethanol (**red**) at various pressures.

D: Ethyl acetate in solvent of distilled water (**black**), solvent of distilled water and 12 % ethanol (**red**) at elevated pressure and red data from A (**green**).

4. Detection of Ethyl Acetate and the effect of Ethanol

The response due to ethyl acetate was isolated through preliminary testing. Following this a stock of distilled water spiked with ethyl acetate was successively diluted so that a range of concentrations could be tested. It was found the system had a very good sensitivity to ethyl acetate and reliable readings thirty times below the human threshold were recorded (A).

The same procedure was undertaken with wine as the solvent. It was found the sensitivity was severely impaired. It appeared that ethanol, due to its abundance within wine, was acting as a co-solvent and depleting the reservoir of reactant ions required for detection. This was tested by creating a solvent of distilled water and 12 % ethanol. The result was an identical response to when the solvent was wine. Sensitivity was improved by substituting the non-polar GC column with a greater polarity. As ethanol elutes from the GC column before ethyl acetate increasing the separation time between the compounds should reduce the depletion of reactant ions. This was found to be the case (A). The analysis time however increased and saturation of the reactant ions was still observed.

5. Recovery of lost signal and Conclusions

A method to overcome the loss of sensitivity while not increasing the analysis time was sought. The answer required increasing the pressure of the make-up flow of air through the FAIMS device. Due to the high frequency of the electric waveform used within the Owlstone device a phenomenon known as clustering [13] is unlikely. However, increasing the pressure promotes interaction of constituents (and therefore clustering) which increases the separation of ion species within the FAIMS device (B). As the separation of ions is increased through the pressure the strength of the applied waveform can be decreased resulting in a larger reservoir of available reactant ions for analysis. This led to improved sensitivity of ethyl acetate even within a complex solvent such as wine (C).

It has been found that a GC-FAIMS system is capable of detecting ethyl acetate within wine at levels far below the human perception threshold. It has also been shown that a FAIMS device can be tailored to account for a complex background without increasing analysis time or resources.

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